

IN Patent No. 81845 IN

Chenks

THE PATENTS ACT, 1970

cont.

COMPLETE
SPECIFICATION

SECTION 10

Patent

TITLE

A process of preparing a bio-pesticide neem
extract

10/10/70

4935435

APPLICANT

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Madras-1
an Indian Company

① Explanation

② Report

③ Gazette: 1 Nov 1970

Application: 1445/MAS/97

The following specification particularly describes
and ascertains the nature of this invention and the manner
in which it is to be performed:

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The invention relates to a process of preparing a bio-pesticide neem extract.

It is known that various parts of neem particularly kernel contain substances with excellent properties for an environment friendly pesticide. Azadirachtin is one of the substances which is most potent and abundant in neem seed kernel.

Methods are known for extracting substances having pesticidal effects from neem seed kernels. In some known methods the kernels are crushed and then either oil is removed first and then the substance containing azadirachtin and other active compounds are extracted. In the process of removing oil, the properties of some heat sensitive neem ingredients, useful as pesticides are affected.

The invention provides a process overcoming these disadvantages. The process according to the invention do not crush the neem seed kernels. Three major steps are used, namely solid-liquid extraction, liquid-liquid extraction and purification. Throughout the process the temperature is not allowed to increase more than 45°C.

The solid-liquid extraction is carried out in an extractor with a counter current flow of the extraction solvent. The extraction solvent used is a saturated aqueous solution of polar organic solvents partially miscible with water and selected from ketones, alcohols, esters and chlorinated hydrocarbons. After sufficient time, the extraction solvent is separated and allowed to settle into two layers. The bottom layer will be aqueous phase and the top layer will be the lighter phase containing polar organic solvent with the active substance.

The top layer is separated and centrifuged with the addition of polar organic solvent to obtain a clear solvent extract. This solvent extract is evaporated at a temperature lower than 45°C preferably in a wiped film evaporator under reduced pressure. The condensate from the wiped film evaporator is further dried at a temperature of less than 45°C. at reduced pressure and then washed with non-polar organic solvents such as hexane, petroleum ether or benzene and dried to obtain a neem extract containing at least 20% of azadirachtin.

Thus the invention provides a process of preparing a bio-pesticide neem extract containing at least 20% azadirachtin, said process comprising the steps of flowing a saturated aqueous solution as an extraction solvent counter currently through uncrushed neem seed kernels, the said saturated aqueous

solution containing non-polar organic solvents partly miscible with water selected from ketones, alcohols, esters and chlorinated, hydrocarbons; allowing the solvent extract to settle into two layers comprising a top lighter phase and a bottom heavier aqueous phase; separating the lighter phase and recycling the aqueous phase to the first step; mixing the lighter phase with additional polar organic solvent and centrifuged to obtain a clear solvent extract free of heavy phase; the clear solvent extract is evaporated at a pressure lower than the atmospheric pressure, maintaining the temperature of the extract to less than 45°C and the residue is further dried at a pressure lower than atmospheric pressure and at a temperature lower than 45°C to obtain a wet substance; the wet substance is dissolved and precipitated in a solvent selected from hexane, petroleum ether and benzene, and the precipitate is dried in vacuum to obtain pure neem extract containing at least 20% azadirachtin.

The process will be further described with reference to the accompanying drawing showing the process flow.

The neem seed is subjected to a counter current flow of a saturated aqueous solvent after initial extraction with intermittent agitator in the aqueous solvent containing a mixture of water and a polar organic solvent selected from ketones,

alcohols, esters and chlorinated hydrocarbons. The polar organic solvent used are only partially miscible with water. Preferred solvents are ketones such as butanone, pentanone and hexanone, alcohols such as butanol and pentanol, and chlorinated hydrocarbons such as chloromethane and trichloromethane. Preferred pH of the extraction solvent is from 2 to 9. The solvent extract from the solid-liquid extraction step is allowed to settle, and the lighter phase is separated and liquid-liquid extraction is carried out using the same type polar organic solvent used in the first step. The solvent extract is then evaporated at a temperature less than 45°C and at a pressure below the atmospheric pressure. It is preferable to use wiped film evaporator to avoid the rise of temperature of the extract above 45°C. After evaporation the residue is further dried at low pressure and at a temperature not exceeding 45°C. It is then purified by dissolving and precipitating in an organic solvent selected from hexane, petroleum ether and benzene. The precipitate is dried in vacuum to obtain the bio-pesticide of neem extract containing at least 20% azadirachtin.

The process according to the invention provides bio-pesticide of neem extract with better pesticidal activity compared to neem extracts produced by process known in prior art due to the higher azadirachtin content and the purity of the extract.

WE CLAIM :

1. A process of preparing a bio-pesticide neem extract containing at least 20% azadirachtin, said process comprising the steps of flowing a saturated aqueous solution as an extraction solvent counter currently through uncrushed neem seed kernels, the said saturated aqueous solution containing non-polar organic solvents partly miscible with water selected from ketones, alcohols, esters and chlorinated, hydrocarbons; allowing the solvent extract to settle into two layers comprising a top lighter phase and a bottom heavier aqueous phase; separating the lighter phase and recycling the aqueous phase to the first step; mixing the lighter phase with additional polar organic solvent and centrifuged to obtain a clear solvent extract free of heavy phase; the clear solvent extract is evaporated at a pressure lower than the atmospheric pressure, maintaining the temperature of the extract to less than 45°C and the residue is further dried at a pressure lower than atmospheric pressure and at a temperature lower than 45°C to obtain a wet substance; the wet substance is dissolved and precipitated in a solvent selected from hexane, petroleum ether and benzene, and the precipitate is dried in vacuum to obtain pure neem extract containing at least 20% azadirachtin.

2. A process of preparing a bio-pesticide neem extract, substantially, as hereinabove described and illustrated with reference to the accompanying drawing.

Dated 8th day of November 1995

(HVG MENON)
OF DePENNING & DePENNING
AGENT FOR THE APPLICANTS

A B S T R A C T

The invention relates to a process of preparing a bio-pesticide neem extract containing at least 20% azadirachtin. The process comprises the steps of flowing a saturated aqueous solution as an extraction solvent counter currently through uncrushed neem seed kernels. The said saturated aqueous solution containing non-polar organic solvents partly miscible with water are selected from ketones, alcohols, esters and chlorinated, hydrocarbons. Then allowing the solvent extract to settle into two layers comprising of a top lighter phase and a bottom heavier aqueous phase. The lighter phase is separated and the aqueous phase is recycled to the first step. Then, the lighter phase is mixed with additional polar organic solvent and it is then centrifuged to obtain a clear solvent extract free of heavy phase. The clear solvent extract is evaporated at a pressure lower than the atmospheric pressure, maintaining the temperature of the extract to less than 45°C and the residue obtained is further dried to obtain a wet substance; the wet substance is dissolved and precipitated and the precipitate is dried in vacuum to obtain pure neem extract containing at least 20% azadirachtin.

POWER OF ATTORNEY

Insert full name,
occupation, address
and nationality of
each applicant

the matter of
Central Act No. 39 of 1970,
and in the matter of

E.I.D. PARRY (INDIA) LTD. of
234, Dare House, Madras-1.
Tamil Nadu, an Indian company;

~~We~~ We the above named applicant(s) do hereby retain, constitute and appoint
Mr. R. G. Depenning of De Penning & De Penning having an office at 10, Government Place
East, Calcutta-700 069, West Bengal and an office at Alaknanda Building, 16, Nepean Sea Road,
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Nadu, and Ms. M A JOSE, R P BHATTCHARYYA, R R NAIR, M V G MENON,
S N MUKHERJEE, DR B L BANNERJEE, M BOSE, A C FERNANDO &
B J SOLOMON

jointly and severally as my/our Agents and Attorneys to apply for and obtain
from the Government of India Letters Patent in respect of an invention for

A process of preparing a bio-pesticide neem extract

and ~~X~~/We authorise them, or any of them, to sign ~~XX~~/our name(s) to such papers and
writings and do such acts, including substitution or revocation, as may be necessary or
expedient and lastly ~~X~~/We request that all official communications now or hereafter relating
to the same may be addressed to them at their office in ~~COO X K O O X X X X~~ Madras and that
they be recognised as ~~XX~~/our authorised Agents in all proceedings incidental thereto and
~~We~~ We hereby confirm all action already taken by them in this matter. ~~X~~We also do hereby
revoke previous authorisations, if any, made in respect of this said matters and proceedings.

Dated this 8th day of November 1995

(B R JAWAHARLAL)

B. R. Jawaharlal
Vice President

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